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(57) Abstract

The present invention discloses a method for obtaining feed pellets. The method comprises the addition of active ingredients to feed pellets after the pellets have been extruded. The method further comprises the addition of a solution or suspension of the desired feed or ingredient in a water or oil phase to the pellets under reduced pressure and subsequently increasing the pressure. The method results in pellets with a high degree of loading and in which the active ingredients are homogeneously distributed.

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Method for the preparation of feed pellets

Field of the invention

The present invention relates to a method for preparing feed pellets. The process for the addition of sensitive ingredients during the preparation of pellets is so altered that activity losses are minimized. Specifically, the pellets are treated by mixing a suspension or solution of active ingredients in a fluid phase (for example oil or water) under appropriate conditions with carrier material which has been previously granulated.

Background of the invention

Feed is generally prepared by mixing different ingredients which are found to be necessary (active ingredients) with carrier materials essential to obtain the feed in the desired form. The desired form may be a powder, a pellet, a solution or a suspension. The preferred form will depend on the application conditions, the composition and the transport.

A well known problem in feed preparation is the loss of active substance during the pelleting process, especially when extrusion is used. Extensive research results have been published describing the loss of activity of the active ingredients during extrusion cooking.

Lee et al. (AIChE Symposium Series (1978) 172 : 192-195) investigated the stability of vitamin A in extrusion cooking processing. The percent of retention of the different tested forms of vitamin A ranged from 50 to 100%. This was reported to be relatively stable when compared with the retention of cantaxanthin which was reported to be in the range of 30 to 35%. Berset (Ind. Aliment. Agric. (1987) 104 : 529-533) reports a loss of 18% of cantaxanthin when a

commercial preparation was treated. Hencken and Estermann (Aquaculture Ind. Develop. Report (1991) 91 : 34-51) report a 25 to 48% loss of activity of synthetic astaxanthin during fish feed preparation using extrusion cooking.

A possible solution to this loss of active substance during granulation is to add the active ingredient in a suitable form like a suspension or solution in a fluid phase subsequent to the granulation step. Mixing of components with a fluid phase can be difficult since active ingredients are not always soluble. Furthermore, the addition after the extrusion step makes it more difficult to obtain a homogenous product.

High concentrations of active ingredients in feed for cattle, poultry or fish in a form which is easy to handle and easy to quantify can be obtained by soaking granulated particles with a suspension or solution containing the desired feed or ingredients thereof. The granulated material contains pores which are filled with the solution or suspension and the loaded pellets can easily be used.

The loading of the pellets is generally performed at atmospheric pressure. The process poses some severe drawbacks:

- it is difficult to reproduce the amount of suspension or solution which is absorbed into the pellets, this is especially relevant if the aim is to control the amount of fluid (oil etc.) which is fed to the animal concerned,
- the pellets are generally preferentially covered at their surface, which may cause problems (like oxidation) of the active ingredient,
- the particles in the suspension may block the pores leading to an inefficient use of the particle pore volume and to the above mentioned surface phenomena.

There is thus a lack of reproducibility in dosage of the active substance and a higher chance to losses of active ingredient as a consequence of insufficient penetration of the active ingredient in the feed granule.

To avoid these problems granulated material with wide pores can be used but this evidently leads to loss of strength of the loaded pellets.

The present invention overcomes the problems described above.

Summary of the invention

The present invention discloses a method for minimizing the loss of activity of sensitive materials due to the well known extrusion process. The invention discloses that this can be done by addition of the material after the extrusion process. The invention is exemplified by the addition of astaxanthin in oil to feed pellets. The invention thus discloses a method for preparing feed pellets loaded with an active ingredient comprising the addition of active material after the extrusion process. The active ingredient is an enzyme, a vitamin, a pigment or a carotenoid. Preferably, the active ingredient is astaxanthin.

The present invention further provides a method for preparing pellets which are homogenously loaded with active ingredient. The method comprises the addition of a suspension or solution of the active ingredients to pellets under reduced pressure and subsequently increasing the pressure. Preferably, the addition is performed under vacuum.

The present invention also provides feed pellets which have been obtained by this method. Specifically, fish meal pellets soaked with an oil suspension containing astaxanthin are disclosed. Such pellets can be recognized by their high degree of loading which is moreover relatively homogenous. A fish pellet is disclosed which contains more than 81.5 mg astaxanthin per kg dry matter.

Detailed description of the Figures

Figure 1 shows the spraydried Phaffia rhodozyma suspended in oil after one (A), two (B) and three (C) passes through a bead mill.

Figure 2 shows the results of the addition of the suspensions of Figure 1 (A), (B) and (C) under atmospheric pressure to the fish meal pellets.

Figure 3 shows the results of the addition of the suspensions of Figure 1 (A), (B) and (C) under vacuum to the fish meal pellets.

Detailed description of the invention

The present invention discloses a method for preparing feed pellets. The present invention discloses how the loss of activity of sensitive materials due to the well known extrusion process can be minimized. The invention discloses a a method for preparing feed pellets loaded with an active ingredient comprising the addition of active material after the extrusion process. The extrusion process can also be the so-called extrusion cooking process. The active ingredient is an enzyme or protein, a pigment or a carotenoid.

The addition of the active ingredient (or any ingredient which can be damaged by the extrusion process) after the extrusion process may cause a problem due to the fact that the extrusion process also serves to obtain a homogenous feed pellet. This problem can be circumvented by the addition of a suspension or solution of feed or ingredients thereof in a fluid phase to the pellets under reduced pressure and subsequently increasing the pressure. The reduced pressure can be vacuum.

The fluid phase can be any fluid (for example water or oil) provided that the active ingredient can be dispersed or dissolved in this fluid. In the present examples capelin-oil is used.

The present invention discloses a method for preparing pellets loaded in a controlled manner with active ingredients. The method comprises the addition under mixing of a suspension or solution of feed or ingredients thereof in a fluid phase to the pellets under vacuum and subsequently releasing the vacuum.

The pellets are composed of an edible material. The specific composition depends on the desired characteristics of the material. The choice of the pellet, volume, weight and pore diameter also depends on the kind of feed one wants to use and on the application for which the loaded pellets are meant. A preferred pellet material in the present invention is fish meal.

The feed or feed ingredient may be any ingredient that is needed. The choice may depend on the nutritional value or on certain rheological characteristics which may be obtained by the activity of the ingredient. In the following some examples of feed or feed ingredients will be discussed. In general terms enzymes or proteins, pigments, vitamins, antioxidants, colouring agents and carotenoids can be employed. Obviously combinations of these ingredients can be added, simultaneously or successively.

a) Enzymes

In general all feed enzymes can be used in the present method these include phytase, amylase and protease. Amylase may be brought on the pellet and subsequently the pellets can be mixed with feed. It is also possible to add the enzyme suspension after pelletizing/extrusion. The activity of this enzyme can be quantified and the added amount controlled. The enzyme can change the fluidity of the feed to which it is added or improve the digestability of feed components.
Another example of an enzyme is phytase here again the dosage can be controlled by the method of the present invention.

b) Carotenoids

Astaxanthin is used as a natural colourant for salmonids. Astaxanthin cannot be solubilized in a water phase. A suspension of astaxanthin in oil or a suspension of Phaffia rhodozyma cells (or cell fragments) containing astaxanthin in oil, can be used to soak the pellets. Incidentally, it was found that this astaxanthin contrary to the commercial synthetic astaxanthin as used by Hencken and Estermann (1991, opt. cit.) could form a stable suspension in oil.

The amount of oil and astaxanthin fed to the fish can thus be controlled. The control of the amount of oil provides a way of influencing the growth rate of the fish.

Other carotenoids which can advantageously be used in the method of the present invention include ß-carotene, cantaxanthin and zeaxanthin.

Desired pellets are brought under vacuum and the liquid phase which may be a solution or suspension of desired feed or ingredients thereof or other ingredients in a water or oily phase is added. The equipment used can range from a waterjet and a flask which can be vacuated at laboratorium scale via a Rotorvapor to large scale equipment.

The pellets obtained by the present method show a high degree of loading, moreover the pellets are loaded relatively homogenously. Furthermore, the amount of loading can be regulated by regulating the amount of fluid phase added. This is especially important if one wants to regulate the amount of oil provided to the desired animal eg. fish.

The present invention discloses for example fish meal pellets which contain more than 30 mg preferably more than 81.5 mg astaxanthin per kg of dry matter.

The present invention thus provides a method for preparing feed pellets wherein the active ingredient is added after the extrusion. The feed pellets are subsequently loaded

under reduced pressure which makes possible an acurate, reproducible and homogenous loading.

Example I

Preparation of an oil suspension containing astaxanthin

A Phaffia rhodozyma culture was centrifuged and dried by spraydrying. The spraydried powder (d_p= 100 µm) was suspended in a commercial fish-oil (Capelin oil). The mixture of cell material containing astaxanthin and oil was ground in a bead mill. After one pass of milling the particle diameter was determined. Microscopic analysis indicated that a large proportion of the spraydried powder particles were disintegrated. A small portion of the particles (<5%) was unaffected and had a diameter of about 100µm (Fig. 1A). The astaxanthin concentration was about 630 ppm (Suspension I).

A second suspension was prepared (Suspension II) by grinding the particles three times in a bead mill (Fig. 1C). Microscopic analysis revealed that practically all cells were disintegrated, the largest agglomerates of cells had a diameter of less than 20 µm.

The astaxanthin concentration was 330 ppm.

Example II

Soaking of fish meal pellets

Commercial fish meal pellets were obtained from Trouw International (Putten, the Netherlands). These pellets were semi-manufactured, which means that they had not been treated with oil. These pellets have a diameter of 8mm, a length of between 0.8 and 1.2 mm and a weight of between 220 and 330 mg.

Further experiments have all been performed in duplo, data reported are the mean values.

A. Soaking at atmospheric pressure

100 g of fish meal pellets were brought into a 1 L beaker. 25 g of an oil suspension containing about 8 mg astaxanthin, was added (Suspension II or twice diluted Suspension I). Mixing was performed during 1 hour in a Turbula mixer.

B. Soaking under vacuum

100 g fish meal pellets were dried under vacuum for 30 min. at 60°C in a 500ml flask in a Rotorvapor. Vacuum was obtained using a waterjet. 25 g of an oil suspension containing astaxanthin was added (as above) in small amounts with vacuum application in between. Rotation was continued for 10 min. and the flask was subsequently cooled on an ice bath.

Example III

Analysis of soaked pellets

A. Microscopic analysis

When pellets were prepared at atmospheric pressure the pellets obtained using Suspension I mainly gave a red colour (astaxanthin) at the surface (Figure 2 A). Pellets obtained using Suspension II showed a red colour also in the interior but the concentration at the surface was higher (Figure 2 B and C).

When pellets were prepared under vacuum Suspension I gave pellets with red colour in the interior and Phaffia mainly at their surface (Figure 3 A). With Suspension II the red colour was mainly in the interior of the pellet (Figure 3 B and C).

Reduction of particle size and application of the Suspension under vacuum clearly gives a better reproducible result.

B. Washing test

The release of astaxanthin from the particles was simulated by washing the pellets. The amount of astaxanthin was determined using HPLC.

Experiments were performed as follows.

a) 50 g fish meal pellet was brought in a 250 ml flask,

b) 100 ml water of 10°C was added,

c) the flask was put in a Shake Water Bath at 10°C, 75 strokes per min. 4.5 cm amplitude for 5 min.

d) after shaking the particles were immediately sieved over a sieve with holes of 1mm diameter,

e) the astaxanthin concentration was determined in untreated pellets, washing water and in the washed pellets, using HPLC.

The following amounts of astaxanthin were found.

A. Atmospheric pressure

Table I

Suspension I	dry matter	asta mg/kg	asta mg/kg dry matter	asta loss
Pellets	0.94	73.0	77.6	
Washwater		1.3	1.5	1.9 %
Washed pellets	0.83	66.0	79.5	

Table II

Suspension II	dry matter	asta mg/kg	asta mg/kg dry matter	asta loss
Pellets	0.95	74.8	78.7	
Washwater		0.28	0.4	0.5 %
Washed pellets	0.82	66.9	81.5	

B. Under vacuum

Table III

Suspension I	dry matter	asta mg/kg	asta mg/kg dry matter	asta loss
Pellets	0.94	82.0	86.3	
Washwater		1.5	1.6	1.8 %
Washed pellets	0.86	73.0	84.8	

Table IV

Suspension II	dry matter	asta mg/kg	asta mg/kg dry matter	asta loss
Pellets	0.95	82.4	86.7	
Washwater		0.16	0.2	0.2 %
Washed pellets	0.75	64.2	85.6	

It is clear that the loss of astaxanthin is lower when particle diameter is smaller (Suspension II). And that the loss is further reduced considerably when pellets are prepared under vacuum.

The above examples merely serve to illustrate the principle underlying the invention and in no way are meant to determine the scope of the disclosure.

Claims

1. A method for preparing feed pellets loaded with an active ingredient comprising the addition of active material after the extrusion process.

2. A method according to claim 1 wherein the active ingredient is an enzyme or protein, a pigment or a carotenoid.

3. A method for preparing pellets loaded with an active ingredient comprising the addition of a suspension or solution of feed or ingredients thereof in a fluid phase to the pellets under reduce pressure and subsequently increasing the pressure.

4. A method according to claim 3 wherein the reduced pressure is vacuum.

5. A method according to claim 3 or 4 characterized in that the fluid phase is water or oil.

6. A method according to claim 3 to 5 characterized in that the active ingredient is an enzyme, a pigment or a carotenoid.

7. A method according to claim 6 characterized in that the enzyme is selected from the group comprising phytase, lactase, protease and amylase.

8. A method according to claim 6 characterized in that the carotenoid is selected from the group comprising astaxanthin, ß-carotene, cantaxanthin and zeaxanthin.

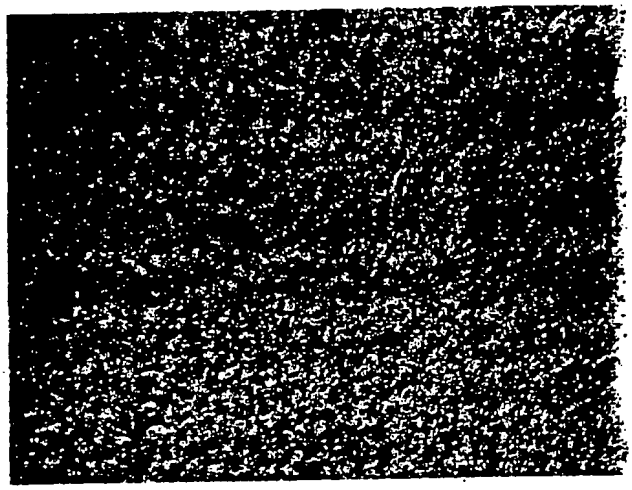
9. A feed pellet characterized in that it contains a high amount of astaxanthin which is homogenously distributed throughout the pellet.

10. A feed pellet according to claim 9 characterized in that it contains more than 30 mg astaxanthin per kg dry matter.

FIGURE 1/3

A

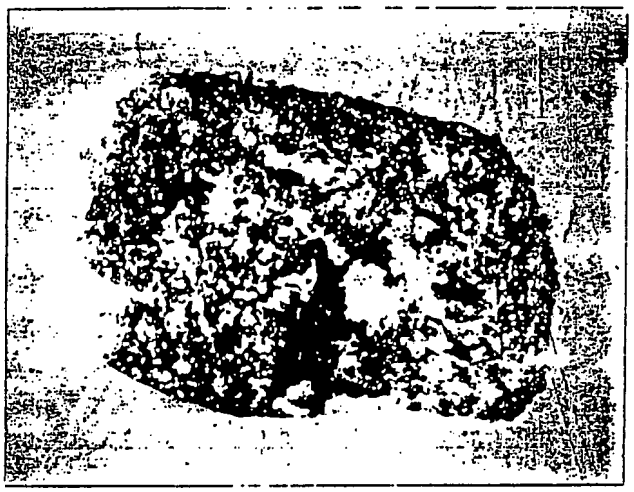
B



C

FIGURE 2/3

A



B

C

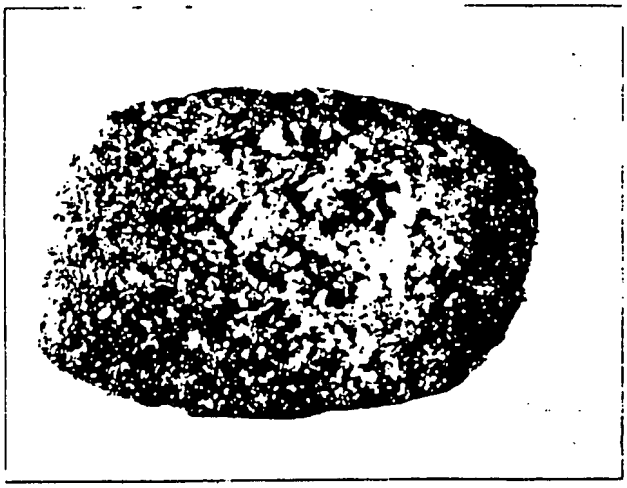
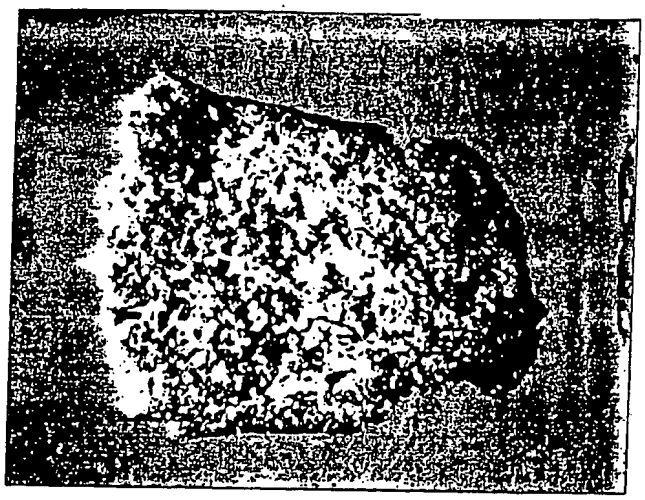
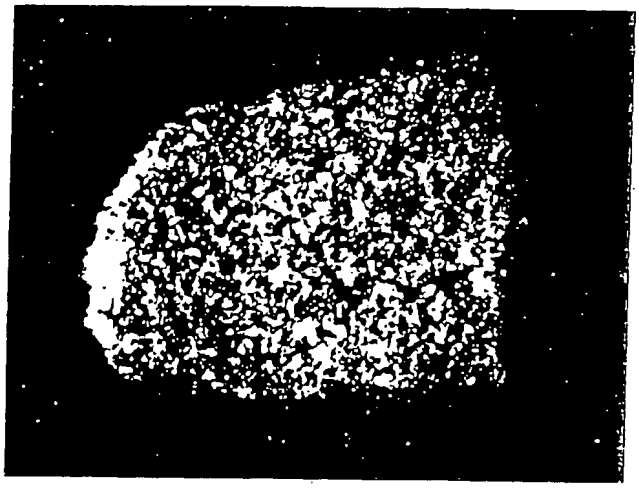


FIGURE 3/3

A



B



C

INTERNATIONAL SEARCH REPORT

International Application No PCT/NL 93/00025

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 A23K1/16

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	A23K

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No.[13]
X	GB,A,1 572 761 (HENKEL) 6 August 1980 see page 2, line 22 - line 34; claims 1-8 ---	1,2
X	GB,A,985 613 (F. HOFFMANN-LA ROCHE) 10 March 1965 see claims 1,3,5; examples 7-9 ---	1,2
X	FEEDSTUFFS vol. 51, no. 3, 15 January 1979, US page 33 JAMES W. ANDREWS ET AL. 'Surface coating of fish feeds with animal fat and ascorbic acid' see the whole document --- -/--	1

° Special categories of cited documents : [10]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
30 MARCH 1993	28. 04. 93
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	DEKEIREL M.J.

Form PCT/ISA/210 (second sheet) (January 1985)

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
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Y	GB,A,2 232 573 (CANADA PACKERS INC) 19 December 1990 see claims 1-14; examples 1-5	3
X	WO,A,8 808 025 (DANISCO BIOTEKNOLOGI A/S) 20 October 1988 see page 19, line 23 - line 28 see page 20, line 21 - page 21, line 2 see claim 24	9,10
A	see example 11	
X	FEEDSTUFFS vol. 49, no. 17, 9 May 1977, US pages 26 - 38 SAMUEL P. MEYERS 'Using crustacean meals and carotenoid-fortified diets' see page 27, column 1; table 4 see page 38, column 1, last paragraph see page 38, column 2, last paragraph - column 3, paragraph 1	9,10
A	WO,A,8 707 116 (JESMA-MATADOR A/S) 3 December 1987 see claim	3
A	DE,A,2 933 261 (HOLZAPFEL) 19 February 1981 see page 7, line 4 - line 35; claims 1,4-7	3

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

NL 9300025
SA 69917

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